Measurement of outgassing rates of signal cables

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Introduction

Due to their importance for LAr purity, we measured the outgassing rates of some materials that are intended to be used for the construction of the ICARUS internal detector. We are interested in particular in the plastics of the signal cables that will constitute the major source of outgassing: depending on the final choice for the cables, in one half-module, a total surface ranging from 180 m^2 to 250 m^2 will be present.

We measured three samples of different cables all produced by Amphenol: high density and normal density twisted pairs cables with no sheath, normal density twisted pairs cables with sheath. The cables were cleaned by washing in ultrasounds bath and then were dried for 50 hours in a vacuum oven at 60 °C. The cables were then left in air covered by an aluminum sheet until their insertion in the vacuum chamber in order to reproduce as much as possible the final working conditions. The characteristics of the three cables samples are here summarized.

Cable 1: High density twisted pair cable, prod. Amphenol, cable with sheathed pieces
(10 cm) every 50 centimeters;
34 x 2 wires;
single wire diameter: Ø = 0.6 mm;
cable length: l = 1300 mm;
approximate surface: $\mathbf{S_{C1} \approx 1500\ cm^2}$
weight: w = 75 g

Cable 2: Normal density twisted pair cable, prod. Amphenol, cable with sheathed pieces
(10 cm) every 50 centimeters; cable with crimped connectors at both ends;
25 x 2 wires;
single wire diameter: Ø = 0.8 mm;
cable length: l = 1050 mm;
approximate surface: $\mathbf{S_{C2} \approx 1300\ cm^2}$
weight: w = 90 g

Cable 3: Normal density twisted pair cable, prod. Amphenol, cable completely sheathed;
25 x 2 wires;
single wire diameter: Ø = 0.8 mm;
cable length: l = 1570 mm;
approximate surface (external surface of the sheath): $\mathbf{S_{C3} \approx 1300\ cm^2}$

weight: w = 140 g

The measurement has been performed in a vacuum chamber already used by us for purity monitor tests. The experimental setup is shown in Figure 1: the vacuum chamber was instrumented with two vacuum gauges (G1 and G2) with an integrated sensitivity range going from 10 mbar to 10^{-9} mbar. A mass spectrometer (S) was used to determine the composition of the desorbed gas. The outgassing rate was measured by closing valve MV1 and measuring the pressure increase in a given time interval (40 seconds).

Setup for Outgassing Measurements

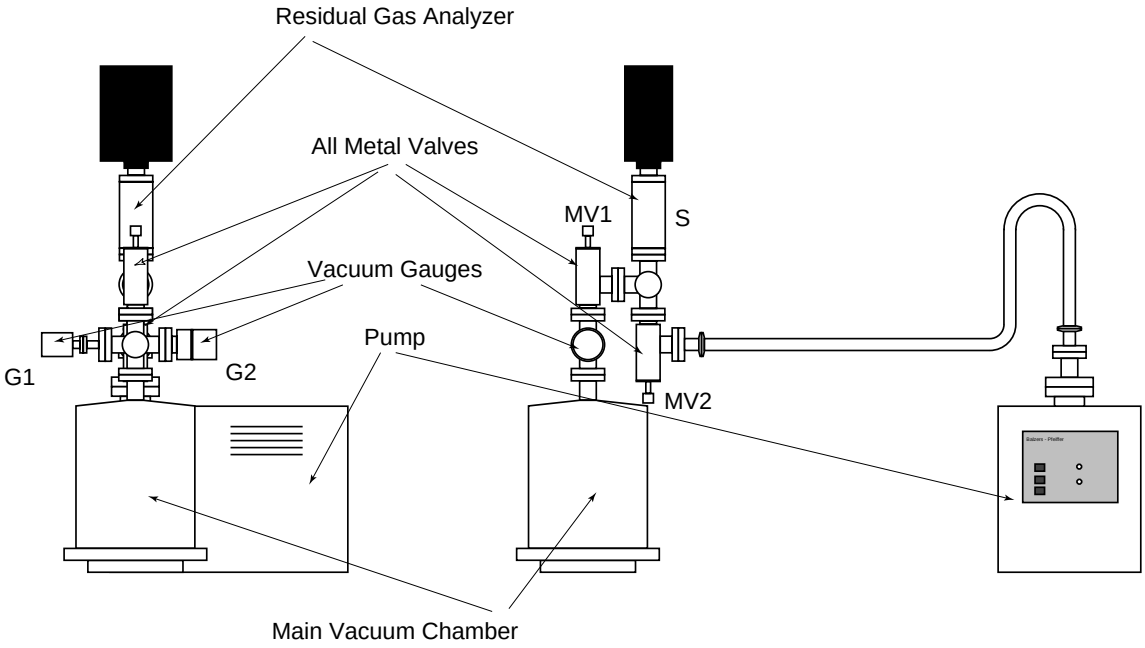

Figure 1: Experimental setup used for the outgassing measurements.

Table 1: Summary data of the experimental setup

Vacuum chamber internal volume (up to valve MV1)	15.6 lt
Vacuum chamber internal surface (up to valve MV1)	3947 cm^2
Turbodrag pump nominal speed	N_2 18 lt /sec He 12 lt / sec H_2 9 lt / sec
Computed conductance of the vacuum line	N_2 0.83 lt / sec He 2.1 lt / sec H_2 3 lt / sec

Estimated effective pumping speed	N_2 0.8 lt / sec He 1.8 lt / sec H_2 2.2 lt / sec
Pressure gauges integrated sensitivity range	$10 \div 10^{-9}$ mbar
Mass spectrometer sensitivity range (mass)	1 - 100 amu
Mass spectrometer sensitivity range (partial pressure)	10^{-6} - 10^{-11} torr

The time interval was chosen in such a way to observe a linear pressure increase while the pump was excluded in order to avoid a systematic outgassing underestimate due to gas re-adsorption by the chamber's or cable's surfaces. The composition of the desorbed gas was measured while the system was under pumping (MV1 and MV2 open).

To subtract the effect of the outgassing of the internal walls of the vacuum chamber, a blank measurement has been performed before the insertion of the cables samples. The chamber has been conditioned with four days of backing under vacuum at about 100 °C. The chamber has then been opened and left in air for 15 minutes to simulate the insertion of a cables' sample. The system has then been evacuated and the internal outgassing has been measured for about five days. The same procedure was used to measure the outgassing of each one of the three cables samples.

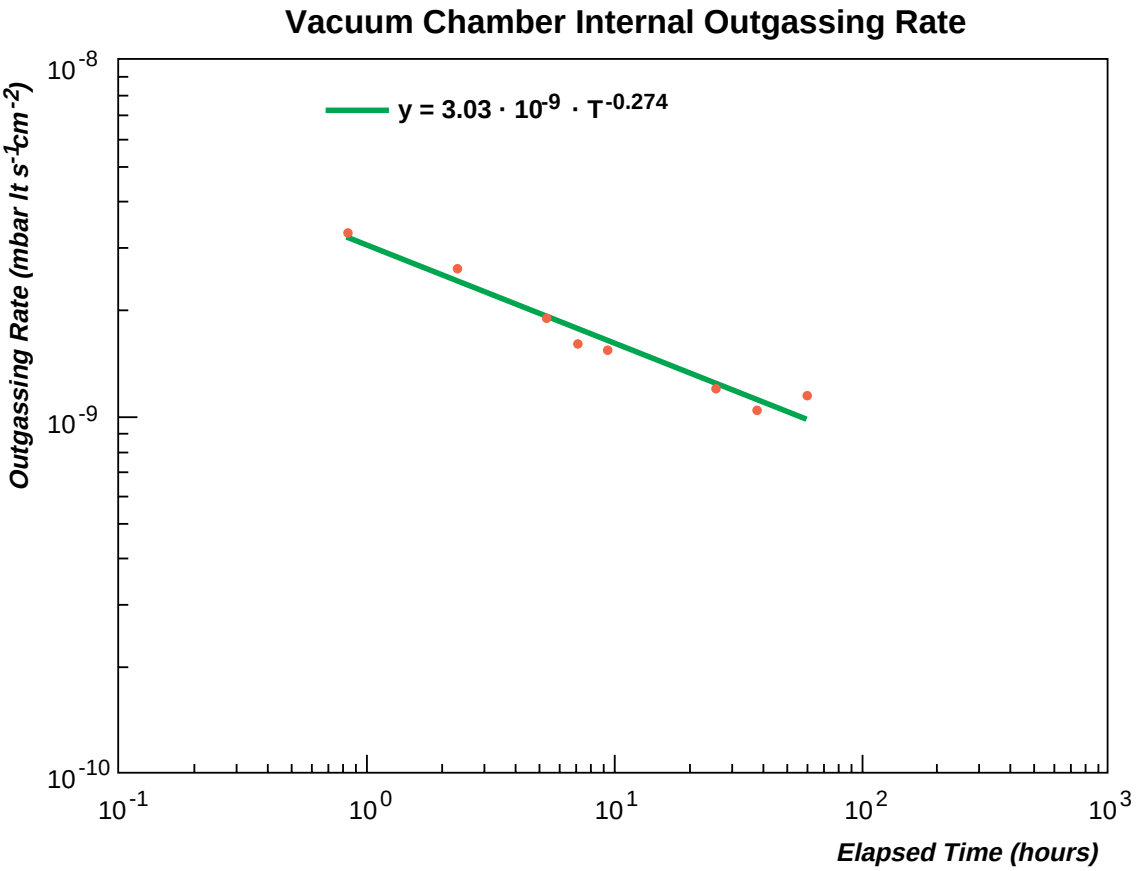

Figure 2: Outgassing rate of the vacuum chamber walls obtained from the blank measurement. The absolute value of the outgassing rate lies in the region usually indicated for electropolished St. Steel.

The result of the blank measurement is shown in Figure 2; it turns out that the outgassing of the chamber walls contributes for a few percent to the total outgassing measured when a cable sample is in the chamber. Notice that the time dependence of the outgassing rate is relatively weak ($\approx T^{-1/4}$); this is probably due to the preliminary conditioning of the vacuum chamber with backing under vacuum at 100 °C. Also, when the pressure approaches the pump limit ($\approx 10^{-6}$ mbar), a saturation effect is expected, that weakens the outgassing time dependence. An outgassing proportional to $T^{-1} \div T^{-0.5}$ is expected under normal conditions.

The desorbed gas from the vacuum chamber (Figure 3) shows the typical composition found in many metals (e.g. st. steel and aluminum). At the beginning, water molecules are primarily desorbed from the surfaces, however, after some hours of pumping, the residual gas composition gets dominated by hydrogen. The other constituents follow the basic composition spectrum of the air but with very different concentrations; oxygen, in particular, is always present in very small percentages (a few percent or less).

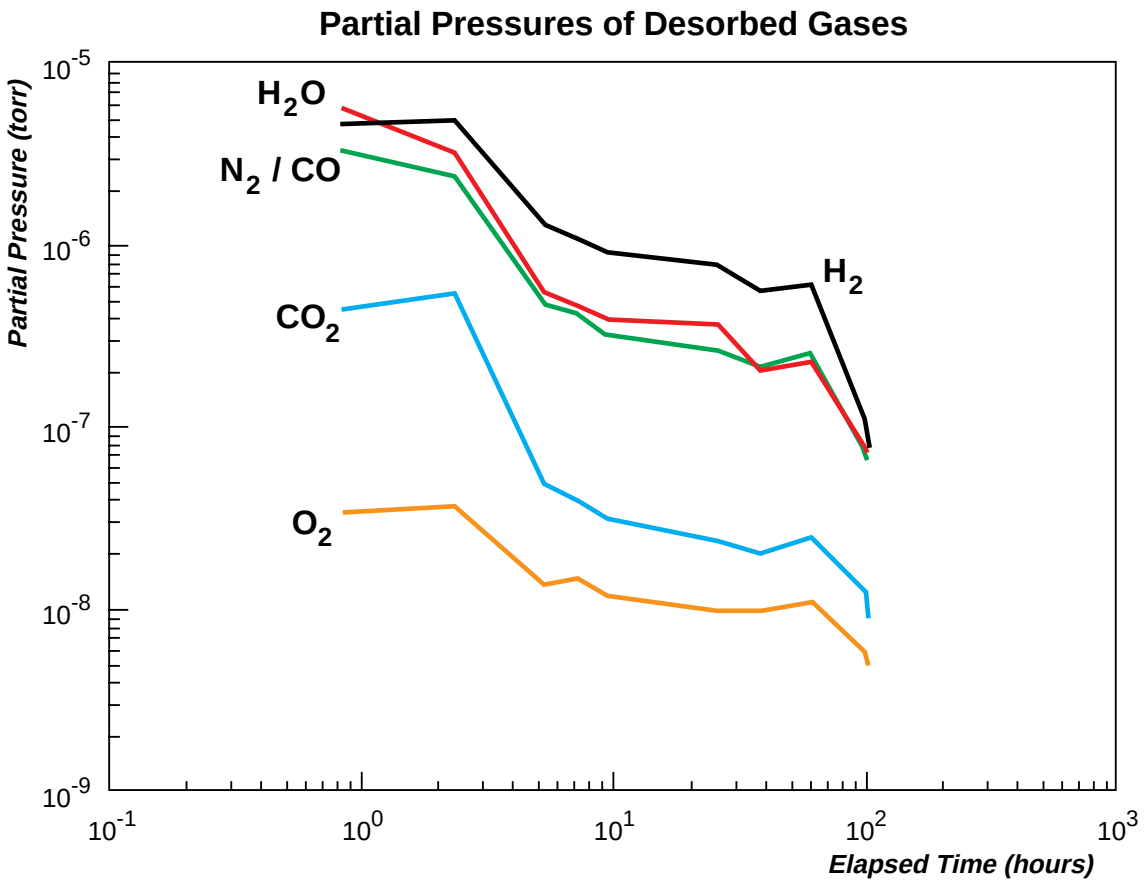

Figure 3: Composition of the gas desorbed by the vacuum chamber walls (blank measurement). Absolute value of the partial pressures should be disregarded as the mass spectrometer was relatively far from the chamber along the vacuum line.

Measurement results

The outgassing rate of the three cables is shown in Figure 4. For all the cables the outgassing rate as a function of the pumping time (T) follows approximately the T^{-1} behavior expected for desorption dominated outgassing[1]. According to the manufactory specifications all three cables are made of the same material (polyolefin: a type of polyethylene); the significant difference in the outgassing rate found between Cable 1 and the other two samples is most likely due to the different exposure time in air. Cable 1 have been left in air for about four days before the measurement while cables 2 and 3 remained exposed for 75 and 89 days respectively. In ICARUS the signal cables will be left in air, mounted on the chambers frame, for few months before the start of the pump down, so our final condition is better represented by Cables 2 and 3.

Somewhat surprisingly the sheat on Cable 3 does not add a significant amount of virtual leaks; we expected in fact a relevant contribution due to

[1] A time exponent of -0.5 is expected for outgassing dominated by diffusion of impurities through the material surface layers

the trapped volumes between the sheat and the single cables or, at least, an enlargement of the effective surface (by a factor larger than 2) with respect to the external one. None of these effects was actually seen. A possible explanation is that the contact surface between the sheat and the cables remains naturally protected when the cable is left in air, and its contribution to the total outgassing becomes negligible with respect to the one from the external surface.

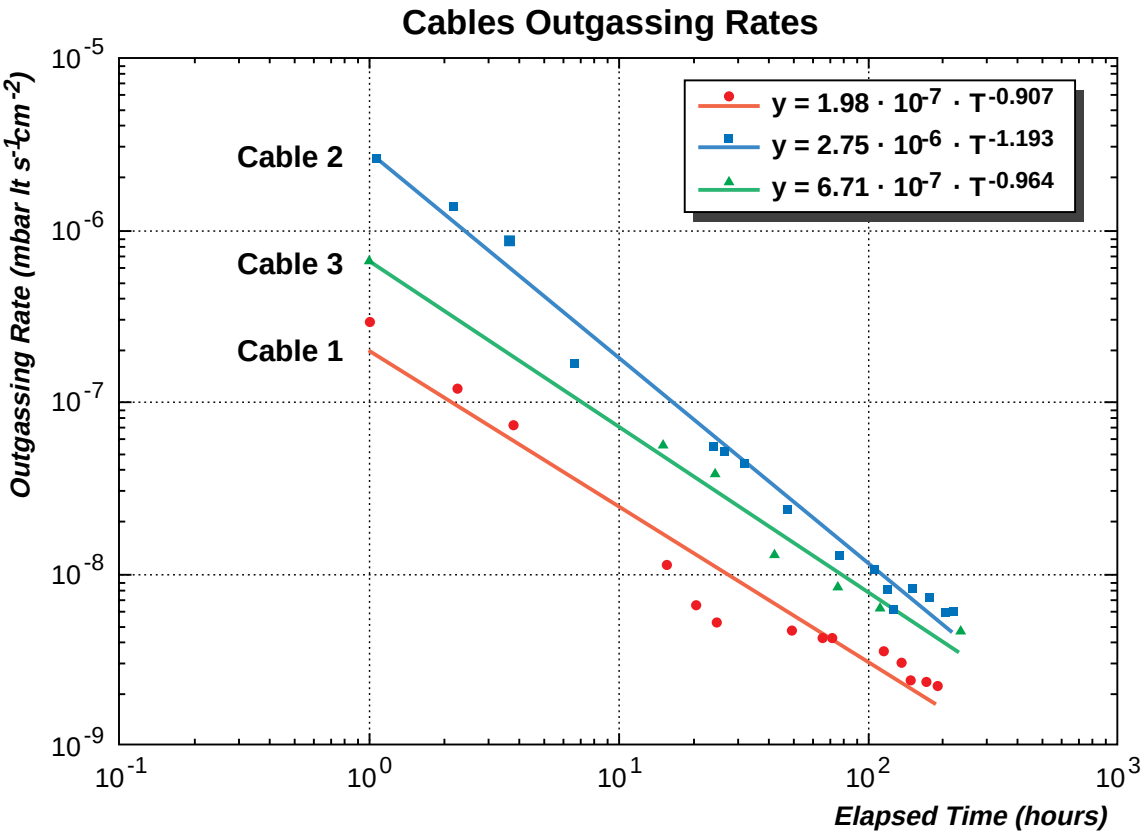

Figure 4: Outgassing rates measured on the three cables samples. Data have been corrected for the internal outgassing of the vacuum chamber.

As a cross-check we can compare the outgassing rate with the base pressure measured while the chamber was under pumping. At equilibrium, pressure and outgassing are related by the formula:

$$P(t) = \frac{O(t)}{S}$$

where S is the effective pumping speed (see Table 1). The comparison is shown in Figure 5 for Cable 2. The good agreement between the two data sets confirms that our measurement method is free from major systematic uncertainties.

For all samples the composition of the desorbed gas is always dominated by water (Figure 6), however, as the pressure decreases, the relative amount of water diminishes and the contribution from other gases (in particular N_2 / CO) becomes important. The interpretation of the gas mixture given by the mass spectrometer software usually reports the

presence of common, light mass, hydrocarbons (Methyl Alcohol, Hexane, Methane, etc.) with a total relative concentration which, at the lowest pressures, can grow up to 20 % ÷ 30 %. These data however have to be taken with some care as they are strongly dependent on the internal instrument calibration and can also be affected by the spectrometer mass limit (100 amu) which could mask the presence of heavier fragments.

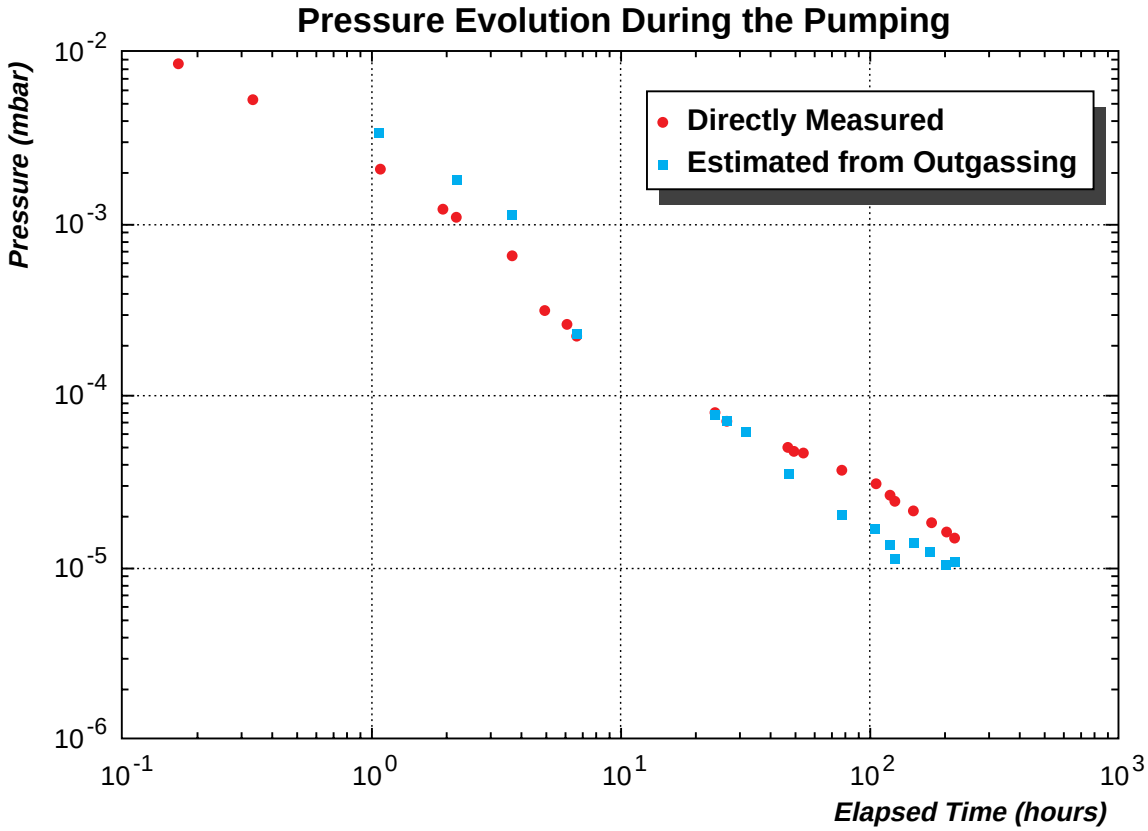

Figure 5: Comparison between the base pressure directly measured in the vacuum chamber during the pumping and the equilibrium pressure due to outgassing calculated with the formula described in the text for Cable 2. Small discrepancies at low pressures are explained by the fact that the pumping speed (for simplicity, assumed constant for the pressure estimate from the outgassing) is decreasing smoothly below 10^{-4} mbar until the limit of the pump ($\approx 10^{-6}$ mbar).

We found a large source of materials outgassing data in the NASA - Marshall Space Flight Center materials test databases (MSFC-SPEC-1443 and MSFC-SPEC-1238)[2]. The outgassing is given there in term of Total Mass Loss (TML) from a sample of material in a vacuum chamber ($\approx 10^{-5}$ torr) backed for 24 hr at 125°C. The data we found on polyolefin insulated wires report a TML going from 0.07 % to 0.28 %. We can infer the total amount of desorbed gas from our cables samples by integrating the outgassing rates in Figure 4:

[2] The databases are freely accessible through the web at the address: http://map1.msfc.nasa.gov/WWW_Root/html/dbase_list.html

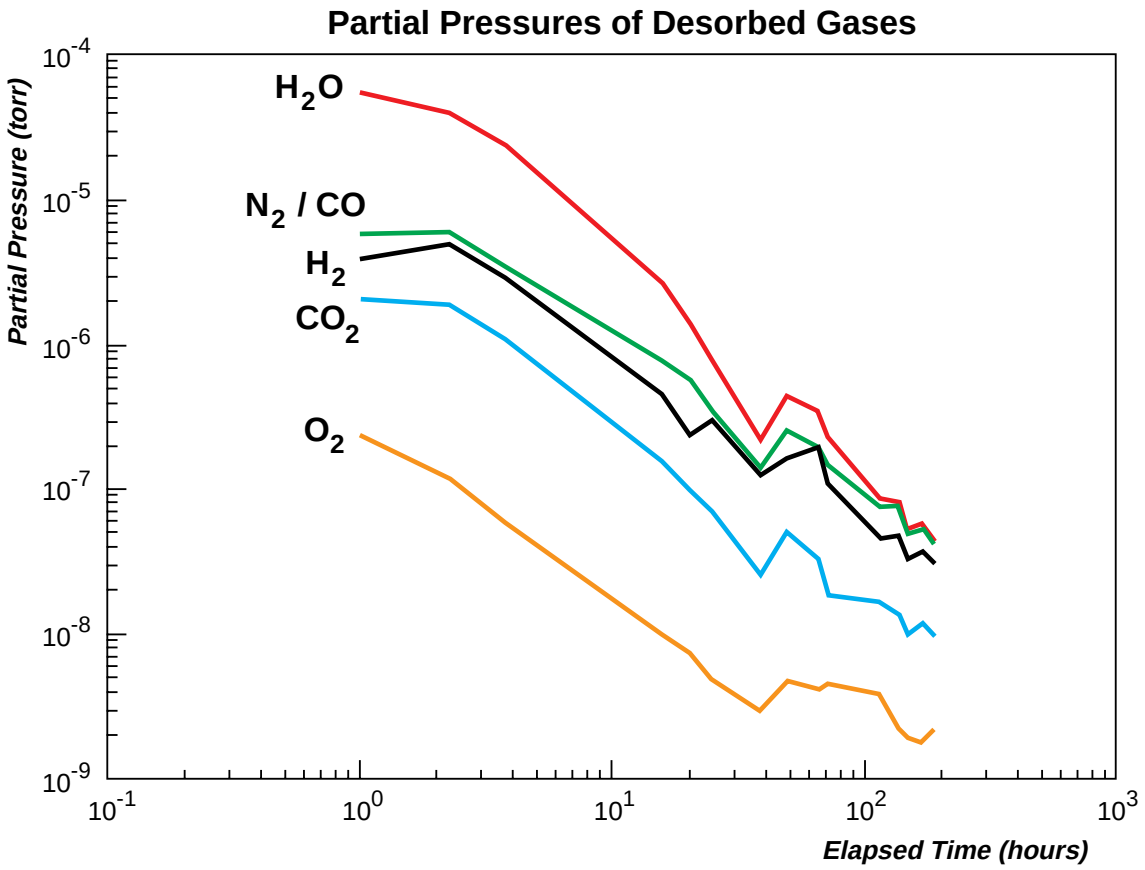

Figure 6: Major components content of the desorbed gas (data from Cable 1) as reported by the mass spectrometer. As already noted (Figure 3) only relative values are meaningful.

$$P \cdot V = \int_{t_1}^{t_2} O(t) \cdot dt = \int_{t_1}^{t_2} \alpha \cdot t^{-\beta} \cdot dt = \frac{\alpha}{1-\beta}\left(t_2^{1-\beta} - t_1^{1-\beta}\right) \qquad \beta \neq 1$$

$$n = \frac{P \cdot V}{R \cdot T}$$

where α and β are deduced from the fits in Figure 4.

Introducing the numerical factors in the equations above (t_1 = 1 hr, t_2 = 200 hr, T = 300K), we obtain for Cable 1, 2 and 3 respectively:

$$n_1 = 3 \cdot 10^{-4}\ mol$$

$$n_2 = 1.7 \cdot 10^{-3}\ mol$$

$$n_3 = 7.5 \cdot 10^{-4}\ mol$$

Taking as the mean atomic weight of the desorbed gas 23 amu (the mean value between water and N_2) we obtain as the Total Mass Loss:

$$TML_1 = \frac{23 \cdot n_1}{w_1} = 0.009\ \%$$

$$TML_2 = 0.044\ \%$$

$$TML_3 = 0.012\ \%$$

Our results compare very favorably with the NASA data indicating that the adopted cleaning and handling procedures are very effective in removing contaminants from the cables surfaces.

We recommend therefore that all cables will be subject to this procedure (washing with an appropriate detergent in ultrasounds, rinsing with demineralized water in ultrasounds and drying at 60 °C in a vacuum oven) and will be stored sealed in a N_2 or Ar filled bag until the last moment before their installation on the wires chambers.

Conclusions

We measured the outgassing rates of three samples of cables foreseen to be used for the internal cabling of the T600 detector.

We found that, even after a long period of exposure to air, the outgassing is relatively modest, especially if compared with other measurements on the same type of material. This indicates that the adopted cleaning and handling procedures are very effective in removing impurities from the cables surfaces.

According to our data, after 100 hours of pumping at molecular regime, the outgassing rate in a T600 half-module (180 m^2 of cables) should be, in the worst case, of the order of 7×10^{-8} mbar sec^{-1}. This would lead to a contamination of impurities (not all electronegative) in the liquid of the order of 0.3 ppb or less for each hour of free outgassing at room temperature. All these rates appear to be easily manageable with the handles that we will have (surfaces conditioning with GAr, pumps, recirculators) taking also into account the foreseen cooling time (some degrees per hour).